



LIME BASED MORTARS FOR CERAMIC TILE APPLICATION: THE INFLUENCE OF THE LIME, THE USE OF A METAKAOLIN AND THE CURING

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ABSTRACT

Bearing in mind the need to repair ceramic tile facade, that were traditionally applied by a lime-based mortar, an experimental campaign was held in order to characterize over time mortars made with two types of lime - an air lime EN 459-1 CL90-S and a recently classified EN 459-1 NHL3.5. The influence of different binder: aggregate proportions on mortars with air lime, of different percentages of a metakaolin substituting the same weight of air lime or of natural hydraulic lime and of different curing conditions are tested and discussed.

Mortars used to apply glazed ceramic tiles will have a low contact with the carbon dioxide of the environment and the hardening of pure air lime mortars will be difficult. For that reason the addition of a metakaolin on air lime-based mortars can be advantageous. But natural hydraulic lime mortar NHL3.5 have registered higher mechanical strength but comparable capillary suction and drying characteristics to air lime mortars; that is why NHL mortars, without or with a low amount of metakaolin, can also be a possibility to intervene glazed ceramic tile façade when the mechanical resistances are compatible.

KEYWORDS: air-lime, natural hydraulic lime, metakaolin, mortar, curing, laboratorial characterization

1. INTRODUCTION

Ceramic tiles were traditionally applied as wall finishing layers glued with air-lime mortars. Air-lime mortars only harden by carbonation, when the calcium hydroxide reacts with the carbon dioxide of the environment. Due to the low contact with the environment of mortars coated by ceramic tiles – only by thin joints – their hardening is very slow. May be that situation was traditionally solved by the expertise of ancient workmanship but nowadays it can be a problem, not only because of lack of professional knowledge on working with pure air-lime mortars but also because of *in situ* schedules [1].

The use of pozzolans that can react with the calcium hydroxide of air-lime mortars make those mortars able not only to harden by carbonation but also to have a partial curing by hydration, what can facilitate its use for the application of ceramic tile finishing layers. On the other hand the ceramic tile



layer reduces the water drying from the fresh mortars to the environment and that can be an advantage to potentiate the pozzolanic reaction [2]. Metakaolins, obtained by thermal treatment at relatively low temperatures and milling of kaolins – a very abundant clay in Portugal – are a possible of artificial pozzolan to use in air-lime based mortars [3].

Another possibility to apply ceramic tile finishing layers on walls can be the use of natural hydraulic lime based mortars. These types of limes have actually to fulfill distinct formulation requirements due to the new version of standard EN 459-1:2010 [4] (in Portugal, the NP EN 459-1: 2011 version, which is ruling since July 2012); they are no longer able to have additions and high levels of sulphates and need to have a minimum level of calcium hydroxide. In natural hydraulic lime mortars, the curing occurs by hydration of some of the hydraulic constituents but also by carbonation of the calcium hydroxide. If a pozzolan is added, a pozzolanic reaction can also take place – most probably competing with the carbonation - between the silica and alumina from the pozzolan and the calcium hydroxide.

In order to increase the knowledge on the behavior of pure air lime and of pure natural hydraulic lime mortars in comparison with similar mortars with a metakaolin used as an artificial pozzolan, these types of mortars have been prepared and characterized. The testing campaign of these mortars involves mechanical, physical, mineralogical and microstructural characterization, but only a part of this work will be focus here, trying to correlate those characteristics with the application on ceramic tile façade. Aspects related with the workability and the hardening of mortars with the different limes, without or with different amounts of a metakaolin, and in defined curing conditions will be analyzed. Also the mortars characteristics in the harden state will be compared between the use of the different limes, the use of different contents of a metakaolin, the curing in different conditions and the evolution with age.

2. EXPERIMENTAL

2.1. Materials

For the production of mortar samples the following materials have been used:

Binder - A hydrated powder air-lime CL90-S from Lusical (designated by CL) and a natural hydraulic lime NHL3.5 from Secil (designated by NHL), according to the producer already classified as a natural hydraulic lime following the specifications of the recent version of standard EN 459:2010 [4].

Pozzolan - A comercial metakaolin Argical M 1200 S from Imerys (designated by Mk).

Aggregate – A well graduated mixture of three siliceous washed sands.

Table 1 presents the materials characterization. The loose bulk density was determined based on EN 1097-3: 1998 [5], the Mk constitution was presented elsewhere [6] and the other values were obtained from the building lime standard [4]. Fig. 1 presents the particle size distribution of the mixture of sands used as aggregate determined based on EN 933-1: 2012 [7].

Table 1 - Characterization of mortar materials [4, 6]

Materials	Loose Bulk Density [g/cm³]	SO₃ [%]	Ca(OH)₂ [%]	SiO₂ [%]	Al₂O₃ [%]
CL	0,36	≤ 2	≥ 80		
NHL	0,85	≤ 2	≥ 25		
Mk	0,29			54	39
Aggregate	1,46				

2.2. Preparation of mortars, samples and curing

All the mortars with natural hydraulic lime NHL were prepared with a volumetric proportion 1:3 - 1 part of binder and 3 parts of aggregate -, while mortars with air lime CL where prepared with the

same volumetric proportion but also with proportion 1:2. Mortars were prepared with each one of the two limes CL and NHL as binder and sand, without any addition, but also mortars were prepared with two different weight percentages of Mk substituting equal weight of the correspondent lime. Due to the different amount of calcium hydroxide of the limes (with what the Mk will probably react), the weight percentages of Mk substitution was 30% and 50% in air-lime mortars CL and was only 10% and 20% in natural hydraulic lime mortars NHL.

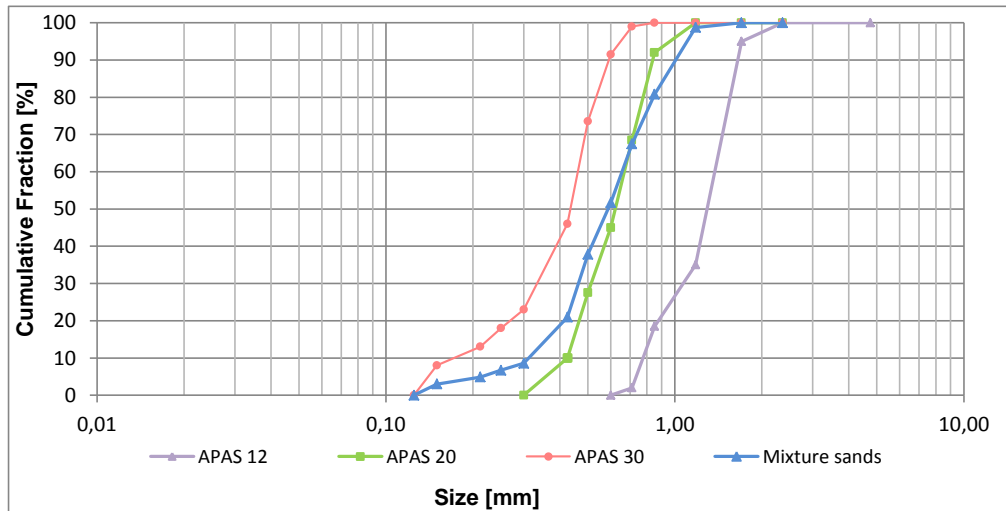


Fig. 1 - Particle size distribution of the sands and of the mixture of sands.

Mortar preparation was based on the EN 1015-2:1998/A1:2006 [8] but some differences were implemented, defined on experimental procedures of project METACAL. The dry constituents were manually homogenized. The quantity of water to achieve a proper workability was previously determined for each mortar and was added during the first seconds of mechanical mixing. The mechanical mixing time was previously defined with a total of 3 minutes. The influence of the water on the fresh mortars was evaluated by the flow table consistency test, based on the EN 1015-3:1999/A1:2004/A2:2006 [9] and results are presented in Table 2. An average value of 151 ± 8 mm was obtained for all workable mortars.

Table 2 presents all the mortars, with its designation – where the type of lime is mentioned, as well as the wt% of lime substitution by Mk –, the volumetric and weight proportion of mortars, the water/binder ratio (considering the binder as the total of lime plus Mk) and the flow table consistency.

Table 2 - Mortar designation, volumetric and weight proportions, water/binder ratio and flow table consistency of mortars.

Mortar	Volum. Prop. (Binder:Agg)	Weight Prop. (Binder:Agg)	Water/Binder [-]	Consistency [mm]
CL_2	1:2	1:8	1,7	149
CL_2_30Mk	1:2	1:8	1,8	154
CL_2_50Mk	1:2	1:8	1,8	151
CL_3	1:3	1:12	2,5	154
CL_3_30Mk	1:3	1:12	2,5	151
CL_3_50Mk	1:3	1:12	2,6	152
NHL	1:3	1:5	1,1	153
NHL_10Mk	1:3	1:5	1,1	149
NHL_20Mk	1:3	1:5	1,1	143

Prismatic 40 x 40 x 160 (mm) samples of all the different mortars were made with metallic moulds, mechanically compacted with 20 strokes in each one of the two layers used to fulfill the moulds. After moulding all the samples remained inside the metallic moulds and closed polyethylene bags. The NHL (without or with Mk substitution) samples were demoulded after two days, while the CL+Mk samples were only demoulded after five days (mortars were too soft at the age of two days). At the fifth day the CL pure samples could not yet been demoulded (mortar remained too soft) and the bags had to be opened for two more days at $65\pm 5\%$ HR and $20\pm 2^\circ\text{C}$ before demoulding.

After being demoulded all the samples were kept inside the polyethylene bags until seven days of age and then placed in contact with three different environmental curing conditions (except the pure air-lime mortar samples which went directly to curing conditions after demoulding): laboratorial humid curing H with $90\pm 5\%$ relative humidity (HR) and $20\pm 2^\circ\text{C}$ temperature; standard laboratorial curing S with $65\pm 5\%$ HR and $20\pm 3^\circ\text{C}$ temperature and natural maritime exposure at LNEC's Cabo Raso, Cascais, station M. Samples stayed at each curing conditions until the age of test. The day before the tests, samples cured at H and M curing were placed at the S environment ($65\pm 5\%$ HR and $20\pm 3^\circ\text{C}$ temperature).

2.3. Testing campaign of harden mortars and results

The testing campaign of the mortars presented in this paper involves dynamic modulus of elasticity, flexural strength, compressive strength, capillary water absorption and drying at the ages of 28 days, 90 days and 180 days (some of the samples are still ageing, reaching 180 days in mid July). Also values of thermal conductivity are presented.

At each age of test the mortar samples of each type (constitution and curing conditions) were used to dynamic modulus of elasticity determination by fundamental resonance frequency, based on EN 14146: 2004 [10], and three points bending flexural strength determination, based on EN 1015-11: 1999 [11]. One half of each specimen from the flexural test were used to compressive strength determination [11].

The half of the samples coming from the flexural test and not used for compression, were dried in an oven at 60°C temperature until constant mass. After cooling in dry environment, they were used for capillary water suction determination (Capillary Coefficient CC in terms of initial capillary suction velocity and Capillary Absorption CAbs in terms of the total of sucked water) [12, 13]. The lateral faces of the samples were watertight by the application of a polyethylene adherent film and the test was held inside a box with saturated environment; the samples were placed over an open grid and the water was maintained with 5 mm high over the base of the samples.

When completely saturated by capillary water, the samples were directly used for the drying test [14, 15], with all the faces watertight (except the top). This situation forced the drying to be unidirectional. During drying the mortar samples were kept in environmental conditions of $20 \pm 2^\circ\text{C}$ temperature and $65 \pm 5\%$ RH.

The average values of a minimum of three samples were used for each test. Results are presented in Tables 3 and 4 for all the mortars and all the testing ages, whenever the test already took place. To the mortars designation presented in Table 2 is added a final letter to identify the type of curing condition (H – Humid, S – following the Standard, M – Maritime exposure).

3. DISCUSSION OF RESULTS

Results of mortars characteristics at 180 days are ongoing and will be able to be presented at the conference. In the meanwhile Table 2 shows the correspondence between the volumetric and the



weight proportions of all the mortars, in terms of binder (lime+Mk) and aggregate. While 1:3 volumetric ratio of air-lime CL based mortars corresponds to 1:12 weight ratio, it corresponds to 1:5 weight ratio for natural hydraulic lime NHL based mortars; 1:2 volumetric ratio of air-lime CL based mortar corresponds to 1:8 weight ratio. Comparing the water/binder ratio between mortars with the two types of lime, it can be seen that it is totally different; the CL based mortar need higher amount of water to achieve comparable consistency and the amount is higher when the binder:aggregate proportion diminishes. It can be remarked that with both types of limes the introduction of Mk diminishes the consistency or implies the need of a higher amount of water.

Table 3 - Mortar/curing volumetric and weight proportions, water/binder ratio and flow table consistency of mortars.

Mortar/curing	Ed [MPa]			FStr [MPa]			CStr [MPa]		
	28d	90d	180d	28d	90d	180d	28d	90d	180d
CL_2_H	1969	3007	3290	0,18	0,36	0,30	0,30	0,62	0,48
CL_2_S	2845	3443		0,32	0,43		0,45	0,79	
CL_2_M	3272	3969		0,32	0,36		0,53	0,64	
CL_2_30Mk_H	6104	6370	6353	0,67	0,65	0,48	2,99	3,61	1,41
CL_2_30Mk_S	5374	6118		0,23	0,18		1,76	1,13	
CL_2_30Mk_M	1624	1089		0,31	0,28		1,83	1,94	
CL_2_50Mk_H	9383	8244	6855	1,13	1,12	1,23	6,33	7,03	3,13
CL_2_50Mk_S	5507	2455		0,57	0,21		2,90	1,24	
CL_2_50Mk_M	3049	3991		0,41	0,42		2,52	2,01	
CL_3_H	1729	3191	3092	0,08	0,24	0,24	0,23	0,39	0,48
CL_3_S	3649	4050		0,36	0,45		0,59	0,70	
CL_3_M	3488	4412		0,28	0,43		0,41	0,65	
CL_3_30Mk_H	4245	1629	5084	0,59	0,16	0,17	0,61	0,48	0,53
CL_3_30Mk_S	1970	2406		0,05	0,05		0,40	0,32	
CL_3_30Mk_M	2047	1699		0,06	0,06		0,40	0,45	
CL_3_50Mk_H	6690	3427	5133	1,09	0,58	0,48	2,53	0,99	1,52
CL_3_50Mk_S	3997	6291		0,12	0,22		0,75	0,93	
CL_3_50Mk_M	3616	1344		0,19	0,10		0,89	0,49	
NHL_H	5181	6982		0,87	1,19		1,51	2,36	
NHL_S	4094	4447		0,52	0,53		1,01	1,22	
NHL_M	4150	6469		0,50	1,06		1,19	2,54	
NHL_10Mk_H	3615	9425		0,75	1,30		3,75	4,02	
NHL_10Mk_S	4951	4256		0,84	0,71		4,07	4,42	
NHL_10Mk_M	5457	5514		0,88	0,84		3,16	4,73	
NHL_20Mk_H	12786	11861		1,39	1,49		7,10	7,64	
NHL_20Mk_S	7746	8382		1,11	0,85		6,93	6,52	
NHL_20Mk_M	8904	7271		1,14	1,13		6,54	5,88	

The hardening of the mortar samples and the capability to be demoulded showed different behaviour between the mortars with the two limes and between the mortars with air-lime CL without and with Mk. It stands out that the addition of Mk to air lime based mortars turns the hardening possible even in environments with high amount of RH and weak contact with CO₂.

Also the type of lime and the existence or not of Mk conducted to differentiated coloured mortars. Air lime based mortars are white and became slightly salmon coloured when Mk is added. NHL based mortars are light grey and the influence of the Mk (in lower percentages) is not visibly noticed.

From Fig. 2 and 3 it can be noticed that, as expected, 1:2 CL mortars present higher strength compared to 1:3 CL mortars. Mortars with air lime with higher percentage of Mk and humid curing register the highest values, similar to the ones registered by NHL mortars. CL mortars at 1:3 and 50%



Mk register a decrease of strength from 28 days. Lower strength is presented by mortars with 1:3 and less humid curing conditions.

Mortars with NHL register higher values of strength (especially flexural strength) with humid curing and higher percentage of Mk.

Table 4 - Mortar/curing capillary coefficient, total capillary absorption and drying index of mortars.

Mortar/curing	CC [kg/m ² min ^{0,5}]			CAbs [kg/m ²]			DI [-]		
	28d	90d	180d	28d	90d	180d	28d	90d	180d
CL_2_H	1,40	1,12	3,5	17,35	18,60	18,5	0,29	0,22	0,24
CL_2_S	2,73	2,81		19,04	18,29		0,41	0,49	
CL_2_M	2,80	2,37		17,57	19,12		0,29	0,39	
CL_2_30Mk_H	1,02	1,12	2,94	26,06	24,80	25,68	0,30	0,30	0,30
CL_2_30Mk_S	2,65	3,28		26,58	26,33		0,42	0,50	
CL_2_30Mk_M	3,35	3,51		26,19	27,09		0,47	0,58	
CL_2_50Mk_H	0,51	0,83	2,15	24,21	22,90	27,83	0,36	0,31	0,36
CL_2_50Mk_S	2,54	2,72		27,55	26,45		0,45	0,48	
CL_2_50Mk_M	3,29	3,11		26,91	26,63		0,45	0,54	
CL_3_H	1,19	0,94	3,46	17,18	18,10	17,96	0,36	0,27	0,27
CL_3_S	2,79	3,00		16,96	17,26		0,31	0,39	
CL_3_M	3,08	2,20		18,06	16,54		0,31	0,43	
CL_3_30Mk_H	2,04	1,38	2,58	22,77	24,10	24,73	0,29	0,35	0,30
CL_3_30Mk_S	4,49	5,15		25,43	25,00		0,40	0,46	
CL_3_30Mk_M	5,35	4,20		25,95	25,31		0,46	0,53	
CL_3_50Mk_H	1,20	1,41	1,87	24,95	26,16	24,36	0,29	0,36	0,28
CL_3_50Mk_S	4,22	4,16		26,09	25,98		0,38	0,55	
CL_3_50Mk_M	3,90	3,16		27,02	25,90		0,40	0,57	
NHL_H	3,01	3,29		23,00	21,90		0,25	0,35	
NHL_S	3,69	4,63		22,41	21,85		0,22	0,32	
NHL_M	3,61	3,62		22,85	21,33		0,24	0,32	
NHL_10Mk_H	2,08	2,69		23,39	21,94		0,36	0,41	
NHL_10Mk_S	2,62	3,12		22,64	22,78		0,28	0,37	
NHL_10Mk_M	2,30	3,02		24,76	21,40		0,31	0,35	
NHL_20Mk_H	1,80	1,80		24,00	18,67		0,47	0,40	
NHL_20Mk_S	2,04	2,22		23,62	22,45		0,38	0,42	
NHL_20Mk_M	1,90	2,47		23,51	21,49		0,41	0,43	

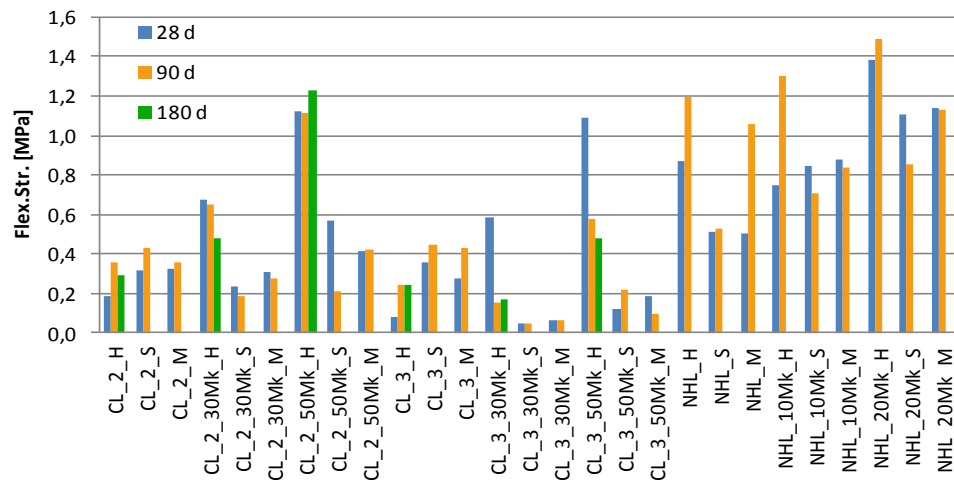


Fig. 2 - Flexural strength of mortars CL_2, CL_3 and NHL at 28, 90 and 180 days.

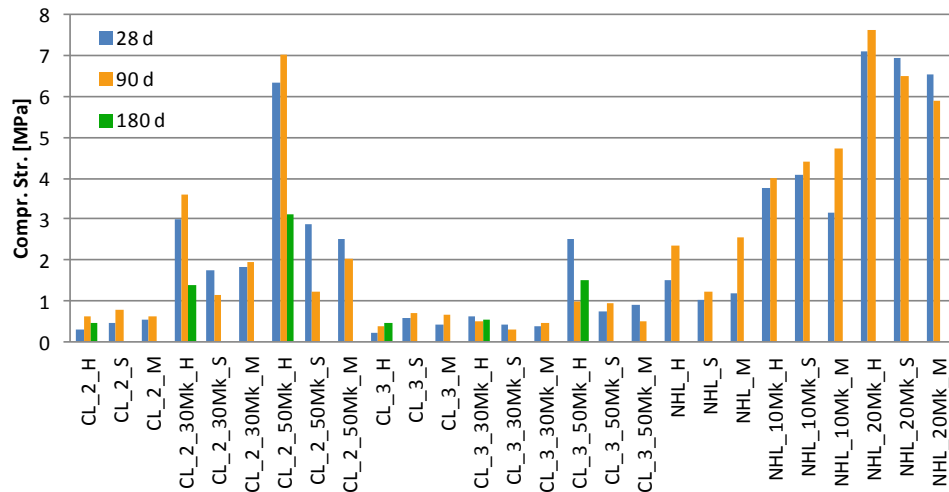


Fig. 3 - Compressive strength of mortars CL_2, CL_3 and NHL at 28, 90 and 180 days.

Some mortars do not present a mechanical resistance increase with time; at young ages the core of the samples are sometimes still humid (particularly for curing with high RH) and that fact can contribute to an increase of mechanical resistances, especially at younger ages. Also the mortars composition (and eventual unstable products that are formed, particularly when Mk is added) can may explain the decrease with age. These situations will be focused in future papers.

From Fig. 4 it can be remarked that the capillary coefficients of mortars CL with 1:2 and 1:3 are similar; there is a slight trend to smaller CC of mortars with humid curing and an inverse trend to higher CC when Mk is added. Among NHL mortars the curing conditions does not seem to influence the results and the addition of Mk seem to diminish CC to values similar to CL mortars.

From Fig. 5 the total capillary absorption of CL mortars seems to be lower without Mk, not being sensible to the curing nor the binder:aggregate proportion. Among the NHL mortars there is no influence of the curing nor the Mk and the registered values are intermediate between the ones registered by mortars CL without or with Mk.

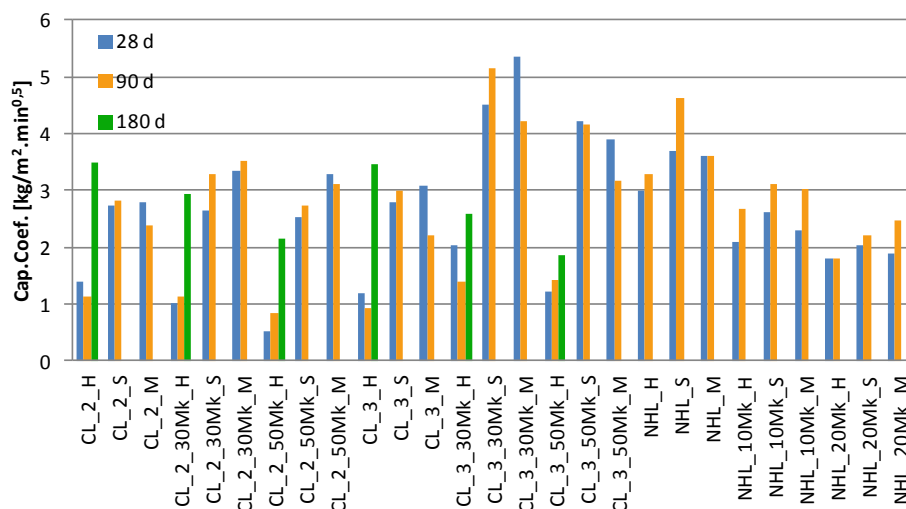


Fig. 4 - Capillary coefficient of mortars CL_2, CL_3 and NHL at 28, 90 and 180 days.

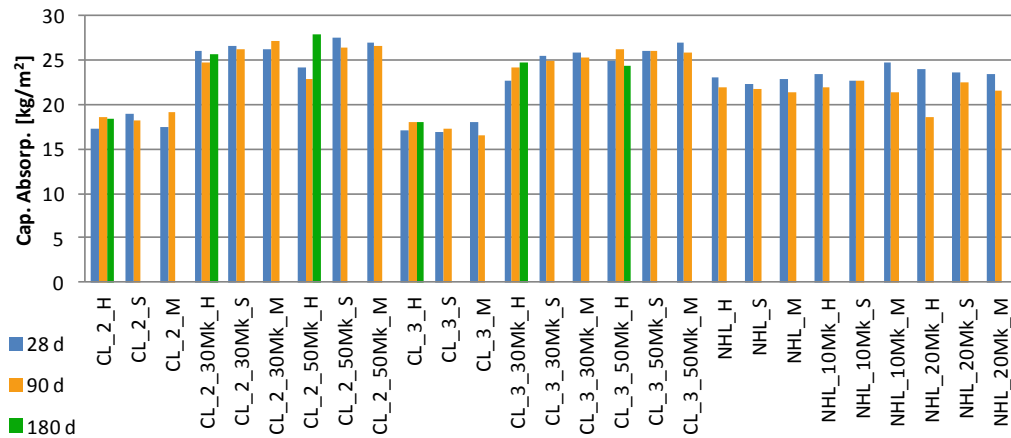


Fig. 5 - Capillary absorption of mortars CL_2, CL_3 and NHL at 28, 90 and 180 days.

Fig. 6 shows that, in CL mortars, less humid curing or the addition of Mk difficult the drying and that similar values of drying index are registered with 1:2 and 1:3 proportions. Among NHL mortars the curing condition does not seem to influence the drying, the addition of Mk slightly difficult the drying but NHL mortars present drying conditions similar (or better) to CL mortars.

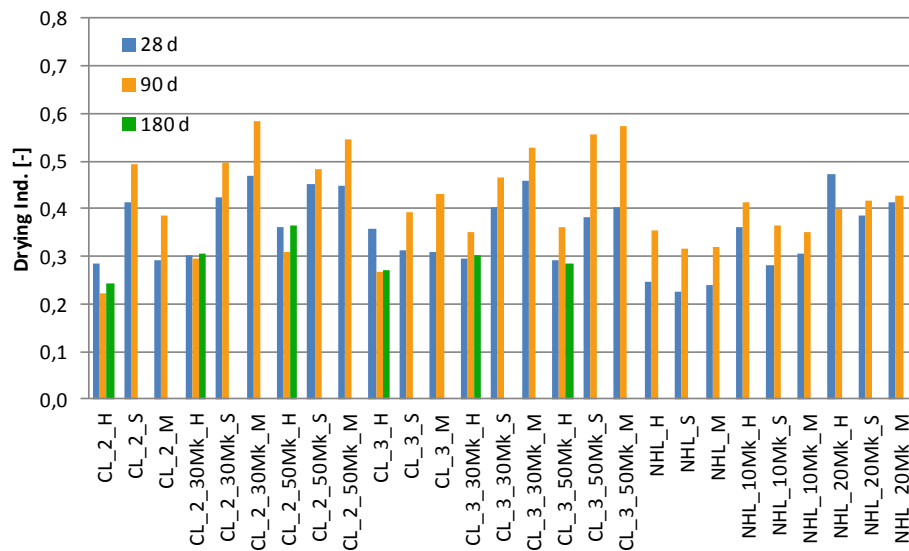


Fig. 6 - Drying index of mortars CL_2, CL_3 and NHL at 28, 90 and 180 days.

Compared to mortars characterized in other experimental works where another natural hydraulic lime was used [16, 17], the NHL used in this work seems more appropriate to be used in ceramic tile adherence layer on ancient walls, especially because of physical compatibility.



4. CONCLUSIONS

Result trends will be confirmed when the 180 days characterization is complete.

The CL based and NHL based mortars characteristics obtained from the experimental campaign show that some of them may be adequate for ceramic tile application. When the option is for an air lime-based mortar, a high amount of metakaolin substituting the lime seems advantageous in terms of hardening and strength without compromising the need of compatibility with the ancient walls [18]. But the option for a natural hydraulic lime mortar can also be a possibility. In this case the use on metakaolin is not indispensable. If the option is for the use of metakaolin, an amount around 10% seems adequate, once higher percentage conducts to probably to high strength mortars for restoration [19]. It should be remarked the physical characteristics of the NHL mortars, comparable to air lime based mortars in terms of capillary suction and drying. This fact allows its application on ancient walls for the repair of ceramic tile façade, whenever mechanical resistances are compatible.

While not directly tested in this study it is expected that the humidity of the fresh mortars that is retained by the glazed ceramic tiles, when compared to other types of applications of mortars, can induce a more humid curing conditions and potentiate the air lime-metakaolin reaction and the natural hydraulic lime mortar characteristics.

5. ACKNOWLEDGMENTS

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